Page 1 16:08 <golam shameem> 05/06/2004

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 NEWS  1          Web Page URLs for STN Seminar Schedule - N. America
 NEWS  2          "Ask CAS" for self-help around the clock
 NEWS  3  JAN 27  Source of Registration (SR) information in REGISTRY updated
                  and searchable
 NEWS  4  JAN 27  A new search aid, the Company Name Thesaurus, available in
                  CA/CAplus
 NEWS  5  FEB 05  German (DE) application and patent publication number format
                  changes
 NEWS  6  MAR 03  MEDLINE and LMEDLINE reloaded
 NEWS  7  MAR 03  MEDLINE file segment of TOXCENTER reloaded
 NEWS  8  MAR 03  FRANCEPAT now available on STN
 NEWS  9  MAR 29  Pharmaceutical Substances (PS) now available on STN
 NEWS 10  MAR 29  WPIFV now available on STN
 NEWS 11  MAR 29  No connect hour charges in WPIFV until May 1, 2004
 NEWS 12  MAR 29  New monthly current-awareness alert (SDI) frequency in RAPRA
 NEWS 13  APR 26  PROMT: New display field available
 NEWS 14  APR 26  IFIPAT/IFIUDB/IFICDB: New super search and display field
                  available
 NEWS 15  APR 26  LITALERT now available on STN
 NEWS 16  APR 27  NLDB: New search and display fields available

 NEWS EXPRESS  MARCH 31 CURRENT WINDOWS VERSION IS V7.00A, CURRENT
               MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
               AND CURRENT DISCOVER FILE IS DATED 26 APRIL 2004
 NEWS HOURS    STN Operating Hours Plus Help Desk Availability
 NEWS INTER    General Internet Information
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 NEWS PHONE    Direct Dial and Telecommunication Network Access to STN
 NEWS WWW      CAS World Wide Web Site (general information)
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FILE 'HOME' ENTERED AT 16:02:44 ON 06 MAY 2004

=> FIL REGISTRY

09889106

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'REGISTRY' ENTERED AT 16:03:14 ON 06 MAY 2004

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 5 MAY 2004 HIGHEST RN 680179-46-8
DICTIONARY FILE UPDATES: 5 MAY 2004 HIGHEST RN 680179-46-8

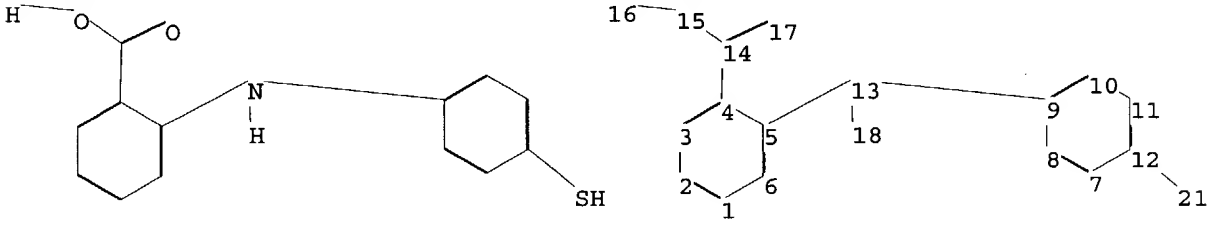
TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2004

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:
http://www.cas.org/ONLINE/DBSS/registryss.html

=>
Uploading C:\Program Files\Stnexp\Queries\10889106C.str



chain nodes :
13 14 15 16 17 18 21
ring nodes :
1 2 3 4 5 6 7 8 9 10 11 12
chain bonds :
4-14 5-13 9-13 12-21 13-18 14-15 14-17 15-16
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 7-8 7-12 8-9 9-10 10-11 11-12
exact/norm bonds :
5-13 9-13 12-21
exact bonds :
4-14 13-18 15-16
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6 7-8 7-12 8-9 9-10 10-11 11-12 14-15 14-17
isolated ring systems :
containing 1 : 7 :

G1:Ak,S

Match level :

09889106

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom
11:Atom 12:Atom 13:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS
21:CLASS

L1 STRUCTURE UPLOADED

=> S L1
SAMPLE SEARCH INITIATED 16:03:30 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 25 TO ITERATE

100.0% PROCESSED 25 ITERATIONS 1 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 200 TO 800
PROJECTED ANSWERS: 1 TO 80

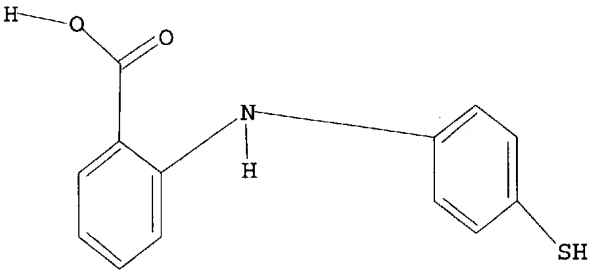
L2 1 SEA SSS SAM L1

=> S L1 SSS FULL
FULL SEARCH INITIATED 16:03:39 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 380 TO ITERATE

100.0% PROCESSED 380 ITERATIONS 9 ANSWERS
SEARCH TIME: 00.00.01

L3 9 SEA SSS FUL L1

=> D L1
L1 HAS NO ANSWERS
L1 STR



G1 Ak,S

Structure attributes must be viewed using STN Express query preparation.

=> FIL CAPLUS

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	155.42	155.63

FILE 'CAPLUS' ENTERED AT 16:03:50 ON 06 MAY 2004
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09889106

FILE COVERS 1907 - 6 May 2004 VOL 140 ISS 19
FILE LAST UPDATED: 5 May 2004 (20040505/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> S L3
L4 12 L3

=> S L4 AND <PY=1999
MISSING TERM 'AND <PY=1999'
The search profile that was entered contains a logical operator followed immediately by another operator.

=> S L4 AND PY<=1999
19722525 PY<=1999
L5 10 L4 AND PY<=1999

=> d l5 ibib abs hitstr tot

L5 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1998:640306 CAPLUS
DOCUMENT NUMBER: 129:261735
TITLE: Water-soluble quinacridone dyes and their use
INVENTOR(S): Etzbach, Karl-Heinz; Kranz, Carolin; Sens, Rudiger
PATENT ASSIGNEE(S): BASF A.-G., Germany
SOURCE: PCT Int. Appl., 19 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

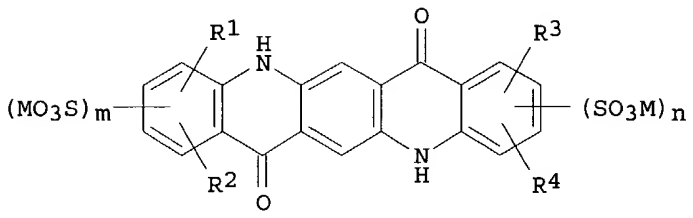
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9841582	A1	19980924	WO 1998-EP1353	19980309 <--
W: JP, US				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
DE 19711443	A1	19980924	DE 1997-19711443	19970319 <--
EP 970149	A1	20000112	EP 1998-913688	19980309
EP 970149	B1	20020828		
R: DE, FR, GB, SE, FI				
JP 2001518129	T2	20011009	JP 1998-540088	19980309
US 6152968	A	20001128	US 1999-380615	19990917

PRIORITY APPLN. INFO.: DE 1997-19711443 A 19970319

WO 1998-EP1353 W 19980309

OTHER SOURCE(S): MARPAT 129:261735

GI



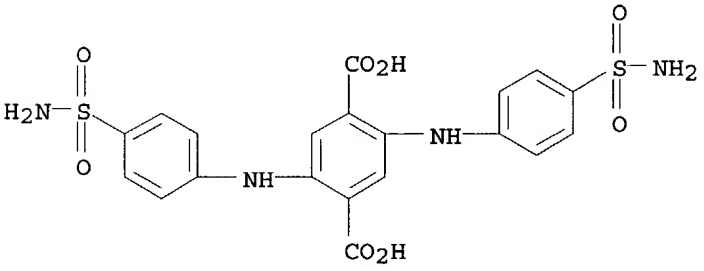
I

AB Water-soluble quinacridones (I; M = Li, K, Na, ammonium; R1, R2, R3, R4 = H, C1-8-alkyl, C1-8-alkoxy, carboxyl, C1-4-alkoxycarbonyl, sulfamoyl, mono- or di-(C1-4)-alkylsulfamoyl, carbamoyl, mono- or di-(C1-4)-alkylcarbamoyl, unsubstituted or substituted mono- or diphenylsulfamoyl, unsubstituted or substituted mono- or diphenylcarbamoyl, halogen, nitro or cyano; m, n = 0-2; sum n + m ≥ 1) and their mixts. are used to dye and print natural and synthetic fiber materials. I may also be used in bulk dyeing of paper and in ink-jet inks and form stable colorant mixts. and wet-fast prints. In an example, 2,5-bis(4-sulfamoylanilino)terephthalic acid was cyclized to 2,9-quinacridonedisulfonic acid, which was obtained in the form of its diammonium salt (λmax 502, 532 nm).

IT **207793-48-4**, 2,5-Bis(4-sulfamoylanilino)terephthalic acid
RL: RCT (Reactant); RACT (Reactant or reagent)
(starting material; water-soluble quinacridone dyes for paper and ink-jet inks)

RN 207793-48-4 CAPLUS

CN 1,4-Benzenedicarboxylic acid, 2,5-bis[[4-(aminosulfonyl)phenyl]amino]- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1998:331458 CAPLUS

DOCUMENT NUMBER: 129:17060

TITLE: Incorporation of sulfonated precursors during quinacridone preparation

INVENTOR(S): Badejo, Ibraheem T.; Britanak, John F.; Rice, Daphne J.

PATENT ASSIGNEE(S): Bayer Corp., USA

SOURCE: U.S., 12 pp.

CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

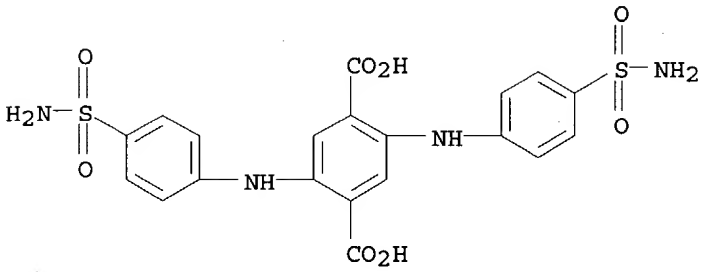
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5755873	A	19980526	US 1996-748742	19961118 <--
CA 2219294	AA	19980518	CA 1997-2219294	19971024 <--
EP 842987	A2	19980520	EP 1997-119395	19971106 <--
EP 842987	A3	19980805		
EP 842987	B1	20020904		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
JP 10158536	A2	19980616	JP 1997-327209	19971113 <--

PRIORITY APPLN. INFO.: US 1996-748742 A 19961118
OTHER SOURCE(S): CASREACT 129:17060; MARPAT 129:17060

AB The first step for preparing quinacridone pigments includes heating a reaction mixture comprising (i) a 2,5-dianilinoterephthalic acid, a 2,5-dianilino-3,6-dihydroterephthalic acid, or a 2,5-dianilino-3,6-dioxo-1,4-cyclohexadiene-1,4-dicarboxylic acid 100, (ii) one or more sulfo- or sulfamoyl-containing derivs. of 2,5-dianilinoterephthalic acid, 2,5-dianilino-3,6-dihydroterephthalic acid, and/or 2,5-dianilino-3,6-dioxo-1,4-cyclohexadiene-1,4-dicarboxylic acid 0.1-15, and (iii) a dehydrating agent 3-20 parts, with the proviso that if either component (i) or component (ii) is a 2,5-dianilino-3,6-dihydroterephthalic acid or derivative thereof, then this step addnl. comprises an oxidation stage. In the second step the reaction mixture from the first step is drowned with a liquid in which the quinacridone pigment is substantially insol. The final step consists of isolating the pigment. The presence of the sulfonated dicarboxylic acid in the ring closure step provides quinacridone pigments having deeper, brighter masstones and improved transparency and rheol. properties. Examples were given for the preparation of quinacridone, 2,9-dimethylquinacridone, and gamma-quinacridone, using polyphosphoric acid cyclization catalyst and 2,5-bis(4-sulfamoylanilino)terephthalic acid, 2,5-bis[4-(3,4-dimethyl-5-isoxazolylsulfamoyl)anilino]terephthalic acid, 2,5-bis[4-(diethylsulfamoyl)anilino]terephthalic acid, or di-Me 2,5-bis[4-(3-methoxypropylsulfamoyl)anilino]-1,4-cyclohexadiene-1,4-dicarboxylate.

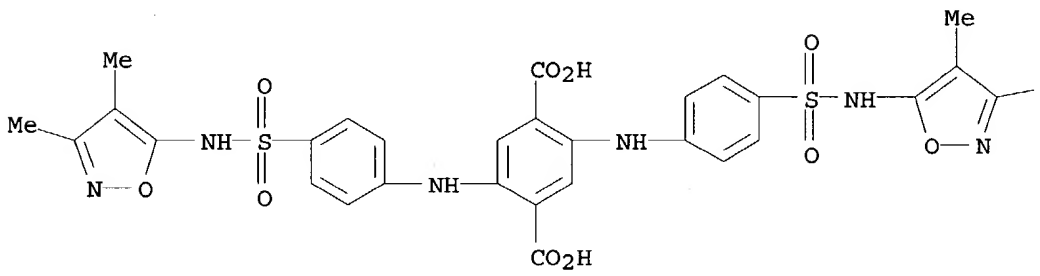
IT **207793-48-4P**, 2,5-Bis(4-sulfamoylanilino)terephthalic acid
207793-52-0P, 2,5-Bis[4-(3,4-dimethyl-5-isoxazolylsulfamoyl)anilino]terephthalic acid
RL: IMF (Industrial manufacture); MOA (Modifier or additive use); PREP (Preparation); USES (Uses)
(preparation of quinacridone pigments in presence of sulfonated precursors)

RN 207793-48-4 CAPLUS
CN 1,4-Benzenedicarboxylic acid, 2,5-bis[[4-(aminosulfonyl)phenyl]amino]-(9CI) (CA INDEX NAME)



RN 207793-52-0 CAPLUS
CN 1,4-Benzenedicarboxylic acid, 2,5-bis[[4-[[(3,4-dimethyl-5-isoxazolyl)amino]sulfonyl]phenyl]amino]- (9CI) (CA INDEX NAME)

PAGE 1-A



PAGE 1-B

—Me

REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

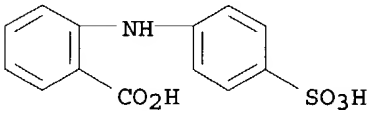
L5 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1993:580482 CAPLUS
DOCUMENT NUMBER: 119:180482
TITLE: Synthesis of N-phenylanthranilic acid using water as solvent
AUTHOR(S): Pellon, Rolando F.; Carrasco, Ramon; Rodes, Lorenzo
CORPORATE SOURCE: Cent. Quim. Farm., Havana, Cuba
SOURCE: Synthetic Communications (**1993**), 23(10), 1447-53
CODEN: SYNCAV; ISSN: 0039-7911
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 119:180482
AB A study of some parameters which influence the Ullmann-Goldberg condensation for the synthesis of N-phenylanthranilic acids was done, showing that these acids can be obtained efficiently using water as the solvent. Thus, 2-ClC6H4CO2H was treated with RC6H4NH2 (R = H, 4-Me, 3-O2N, 3-Cl, 4-H2N, 4-MeO, 4-HO3S) in refluxing H2O in the presence of

powdered Cu to give 9-89% 2-(RC6H4NH)C6H4CO2H.

IT **26119-52-8P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 26119-52-8 CAPLUS

CN Benzoic acid, 2-[(4-sulfophenyl)amino]- (9CI) (CA INDEX NAME)



L5 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1987:489293 CAPLUS

DOCUMENT NUMBER: 107:89293

TITLE: Chloride-channel blockers in the thick ascending limb of the loop of Henle. Structure-activity relationship

AUTHOR(S): Wangemann, P.; Wittner, M.; Di Stefano, A.; Englert, H. C.; Lang, H. J.; Schlatter, E.; Greger, R.

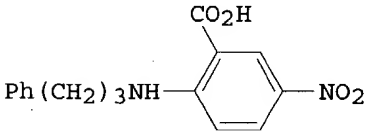
CORPORATE SOURCE: Max-Planck-Inst. Biophys., Frankfurt/Main, D-6000, Fed. Rep. Ger.

SOURCE: Pfluegers Archiv (**1986**), 407(Suppl. 2), S128-S141
CODEN: PFLABK; ISSN: 0031-6768

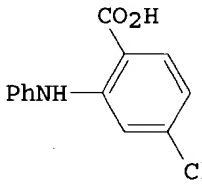
DOCUMENT TYPE: Journal

LANGUAGE: English

GI



I



II

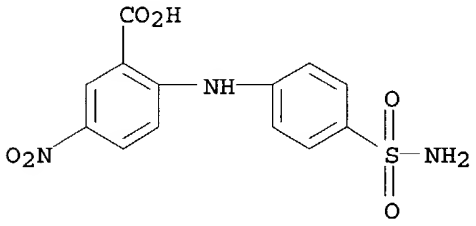
AB On the basis of previous findings with diphenylamine-2-carboxylate a search for compds. which possess an even higher affinity for the Cl--channels in the basolateral membrane of the thick ascending limb of the loop of Henle has been conducted. To quantify the inhibitory potency, measurements of the equivalent short circuit current, corresponding to the secondary active transport of Cl- and measurements of the voltage across the basolateral membrane have been performed. A survey of 219 compds. reveals that relatively simple modifications in the structure of diphenylamine-2-carboxylate led to very potent blockers such as 5-nitro-2-(3-phenylpropylamino)benzoate (I) which inhibits the short circuit current half maximally (IC50) at 8.10-8 mol/L. Structure activity studies suggest that these Cl- channel blockers possess several sites of interaction: The neg. charged carboxylate group, the secondary amine group which probably carries a pos. partial charge, and for the very potent agents (e.g. I and 5-chlorodiphenylamine-2-carboxylic acid (II)) an addnl. neg. partial charge at the resp. -Cl or -NO2 substituent. Finally, also

an apolar interaction with an cycloalkyl or cycloaryl residue seems to be required, and this site of interaction has a defined spacing from the secondary amino N.

IT **107946-91-8P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and chloride channel blocking activity of, structure in relation to)

RN 107946-91-8 CAPLUS

CN Benzoic acid, 2-[[4-(aminosulfonyl)phenyl]amino]-5-nitro- (9CI) (CA INDEX NAME)



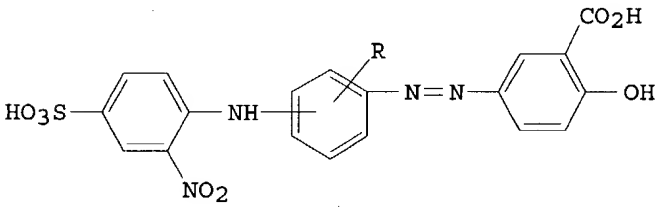
L5 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1981:32168 CAPLUS
DOCUMENT NUMBER: 94:32168
TITLE: Chromium complexes of monozo dyes
PATENT ASSIGNEE(S): Colour-Chem Ltd., India
SOURCE: Indian, 16 pp.
CODEN: INXXAP
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
IN 147672	A	19800524	IN 1977-BO352	19771220 <--

PRIORITY APPLN. INFO.: IN 1977-BO352 19771220

GI

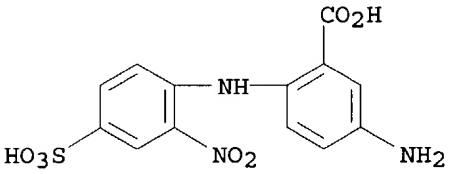


I

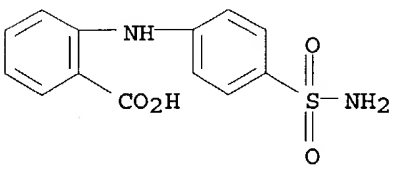
AB Chromium complexes of azo dyes (I; R = H, Cl, Br, Me, Et, MeO, EtO, SO3H, CO2H) are prepared and are used to dye leather fast brown shades. Thus, 4-[(2-nitro-4-sulfophenyl)amino]aniline [135-11-5] was diazotized, coupled with salicylic acid [69-72-7], giving I (R = H, p-substituted) (II) [76091-85-5], which was treated with K chromic sulfate to give Cr complex of II, dyeing leather a fast, dark orange-brown shade. Several addnl. I were similarly prepared

IT **76091-83-3**

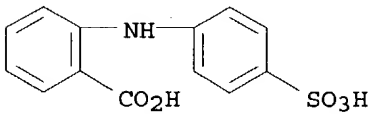
RL: USES (Uses)
(coupling of diazotized, with salicylic acid)
RN 76091-83-3 CAPLUS
CN Benzoic acid, 5-amino-2-[(2-nitro-4-sulfophenyl)amino]- (9CI) (CA INDEX NAME)



L5 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1973:42982 CAPLUS
DOCUMENT NUMBER: 78:42982
TITLE: Syntheses of flufenamic acid metabolites I and II and other N-arylanthranilic acids
AUTHOR(S): Bowman, R. E.; Brunt, K. D.; Godfrey, K. E.; Kruszynska, L.; Reynolds, A. A.; Thrift, R. I.; Waite, D.; Williamson, W. R. N.
CORPORATE SOURCE: Dep. Chem., Parke, Davis and Co., Hounslow, UK
SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (**1973**), (1), 1-4
CODEN: JCPRB4; ISSN: 0300-922X
DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.
AB (Addnl. data considered in abstracting and indexing are available from a source cited in the original document.) 2,5-Cl(HO)C6H3CO2Et reacted with PhCH2Cl-NaOEt-EtOH to give, after hydrolysis, 2,5-Cl(PhCH2O)C6H3CO2H, which was condensed with n-F3CC6H4NH2 in the presence of Cu2+ to give 5-(benzyloxy)-N-(α,α,α-trifluoro-m-tolyl) anthranilic acid (I, R = PhCH2O, R1 = H). Hydrogenolysis gave I (R = OH, R1 = H). 2,5-Cl(O2N)C6H3CF3 was similarly converted into 2,5-PhCH2O(O2N)C6H3CF2; reduction of the NO2 group and condensation with 2-BrC6H4CO2K gave N-[4-(benzyloxy)-α,α,α-trifluoro-m-tolyl] anthranilic acid (I, R = H, R1 = PhCH2O) which gave I (R = H, R1 = OH) on hydrogenelysis. Other N-arylanthranilic acids were prepared by similar Cu- or Cu salt-catalyzed condensations.
IT **39189-35-0P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 39189-35-0 CAPLUS
CN Benzoic acid, 2-[[4-(aminosulfonyl)phenyl]amino]- (9CI) (CA INDEX NAME)



L5 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1970:27990 CAPLUS
DOCUMENT NUMBER: 72:27990
TITLE: Oxidation-reduction indicators for titration in strongly acid media
AUTHOR(S): Bondareva, T. N.; Nikurashina, M. L.; Smirnova, O. A.; Frumina, N. S.
CORPORATE SOURCE: Ural State Univ., Sverdlovsk, USSR
SOURCE: Zhurnal Analiticheskoi Khimii (**1969**), 24(9), 1309-13
CODEN: ZAKHA8; ISSN: 0044-4502
DOCUMENT TYPE: Journal
LANGUAGE: Russian
AB Oxidation-reduction indicators of the diphenylamine class with 2 electrophilic substituents in the mol. were studied. 2-Nitrophenylanthranilic acid (I), 4-sulfophenylanthranilic acid (II), and 2,2'-dicarboxy-diphenylamine (III) can be used as indicators durin g the titration with strong oxidants in a medium of very concentrated H2SO4 and HClO4; titration of Fe(II) with Ce(SO4)2, the titration of Fe(II) wi th dichromate, titrn of oxalates with Ce(IV). The oxidation-reduction potentials of I, II, and III are 0.94, 0.91 and 0.88 V, resp., in 18N H2SO4. The indicators can be used for the determination of polyethylene glycol and polyethoxyamines in aqueous solns. These indicators can be also used for the determination of organic C in soils. To a 0.1-0.2-g soil sample add some crystals of Ag2SO4, then 10 ml 0.2N K2Cr2O7 in 1:1 H2SO4, boil for 5 min, cool, add 2-3 ml H2O and 5 drops of a 0.2% solution of III in a 2% Na2CO3 solution, and after 3-5 min titrate with 0.1N Mohr's salt solution from blue to yellow-green.
IT **26119-52-8**
RL: ANST (Analytical study)
(as oxidation-reduction indicator)
RN 26119-52-8 CAPLUS
CN Benzoic acid, 2-[(4-sulfophenyl)amino]- (9CI) (CA INDEX NAME)



L5 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1965:82557 CAPLUS
DOCUMENT NUMBER: 62:82557
ORIGINAL REFERENCE NO.: 62:14673d-h,14674a-h,14675a-c
TITLE: Phenazines. VI. Synthesis of 2-aminophenazine- and 2-aminocarboxyphenazinesulfonamides
AUTHOR(S): Herbert, R. B.; Holliman, F. G.
CORPORATE SOURCE: Univ. Leeds, UK
SOURCE: Tetrahedron (**1965**), 21(3), 663-75
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 62:82557
GI For diagram(s), see printed CA Issue.
AB cf. CA 60, 6841d, 10678c. Oxidative cyclization of the appropriate aminodiphenylamines, ArAr'NH (I) in boiling PhNO2 yielded R, R1, R2 -substituted 2-aminophenazines (II). The known 2,4-(O2N)2C6H3NHC6H4SO2NH2-

p (500 mg.) hydrogenated 16 hrs. in 50 ml. alc. at 20°/4 atmospheric over 100 mg. PtO2 and the colorless solution filtered into 50 ml. PhNO2, the residue washed with 100 ml. hot PhNO2 and the combined solns. freed from alc., refluxed 36 hrs. and the solution concentrated yielded 46% II (R = 8-NH2, R1 = H, R2 = 2-SO2NH2) (III). Since the SO2NH2 group in III was in conjugation with N-10, a corresponding compound with N-5 conjugation was synthesized. H2O2 (60 ml., 30%) stirred with 11.5 g. 3,4-Br(H2N)C6H3SO2NH2 in 200 ml. AcOH and 4 ml. concentrated H2SO4 and kept 2 hrs. at 70-80° yielded 40% 2,2'-dibromoazoxybenzene,4,4'-disulfonamide, m. 286° (decomposition) (C6H5N). The mother liquor evaporated in vacuo and diluted with H2O yielded 3,4-Br(O2N)C6H3SO2NH2, m. 136-8° (PhMe), triturated (3.25 g.) with 1.8 g. p-H2NC6H4NHAc and 1.5 g. KOAc and the mixture fused 3.5 hrs. at 130-5°, extracted into N NaOH and the decolorized (C) solution acidified with concentrated HCl yielded 48% 4,3-O2N(p-AcNHC6H4NH)C6H3SO2NH2, m. 132-4°, hydrolyzed in 2.5N HCl to the corresponding 4,3-O2N(p-H2NC6H4NH)C6H3SO2NH2 (IV), m. 235-6°, hydrogenated to the diamino compound, 4,3-H2N(p-H2NC6H4NH)C6H3SO2NH2, characterized as the diacetyl derivative, m. 140-2°. IV (500 mg.) hydrogenated and the alc. solution filtered into 150 ml. PhNO2, combined with alc. washings and the alc. evaporated, the PhNO2 solution refluxed 24 hrs. and the filtered solution concentrated yielded 44% II (R = 7-NH2, R1 = H, R2 = 2-SO2NH2) (V), m. 287-9°. In both above syntheses, small amts. of 2-aminophenazine were isolated in addition to III and V. In contrast, an attempt to synthesize II (R = 3-NH2, R1 = H, R2 = 2-SO2NH2) by cyclization of 4,6,3-(H2N)2-(PhNH)C6H2SO2NH2 was frustrated by elimination of the SO2-NH2 group with formation only of 2-aminophenazine. Na2SO3 (10.7 g.) in 200 ml. H2O added in 30 min. with rapid stirring to 20 g. 5,2,4-Cl(O2N)2C6H2Cl in 400 ml. alc. under reflux and the mixture refluxed 2 hrs. with stirring, the filtered solution evaporated and the residue recrystd. from H2O yielded 65% 2,4,5-(O2N)2(Na-O3S)C6H2Cl (VI), m. >300°, refluxed (900 mg.) with 300 mg. PhNH2 and 370 mg. NaOAc in 40 ml. 95% alc. 4 hrs. and the residue on evaporation recrystd. from alc. to give I.2H2O [Ar = Ph, Ar' = 2,4,5-(O2N)2(NaO3S)C6H2]. 3,4,6-Cl(O2N)2C6H2SO2Cl, m. 116.5-17.5°, shaken vigorously (510 mg.) 7 min. in 25 ml. aqueous NH4OH (d. 0.88) and filtered from 18 mg. 3,4,6-Cl(O2N)2C6-H2NH2, m. 175-6°, the orange filtrate evaporated in vacuo and the residue crystallized from alc. yielded 57% 3,4,6-Cl(O2N)2C6H2SO2NH2. The sulfonamide (500 mg.), 400 mg. PhNH2, and 230 mg. NaOAc in 15 ml. alc. refluxed 7 hrs. and the filtered dark red solution cooled yielded 52% I [Ar = Ph, Ar' = 4,6,3-(O2N)2-(H2NO2S)C6H2] (VII), m. 216-18° (alc.). VII hydrogenated and the product acetylated gave I [Ar = Ph, Ar' = 4,6,3-(Ac-NH)2(H2NO2S)C6H2], m. 219-19.5°. VII (140 mg.) hydrogenated and the product refluxed in PhNO2 24 hrs. and chromatographed on Al2O3, the column washed with C6H6 and the orange band eluted with 1:9 Me2CO-Et2O gave 2-aminophenazine. The PhNO2 oxidative cyclization was also successful in the synthesis of 4 selected representatives of the 42 possible 2-amino-carboxyphenazinesulfonamides. The selection was made on the basis of the suggested positions of the CO2H and SONH2 groups in aeruginosin B which behaves similarly to 2-amino- and 2-aminocarboxyphenazinesulfonamides when heated in dilute acid solution p-H2NC6H4SO2NH2 (1.72 g.), 2.91 g. 2,3,5-Br(O2N)2-C6H2CO2H, and 1.64 g. NaOAc refluxed 1 hr. in 30 ml. alc. with stirring and the precipitated yellow Na salt taken up in H2O, acidified with dilute HCl and the precipitate recrystd. from aqueous alc. yielded 63% I [Ar = 2,4,6-HO2C(O2N)2C6H2, Ar' = p-C6H4SO2NH2] (VIII), m. 275-6°, which (500 mg2.) was hydrogenated in 50 ml. absolute

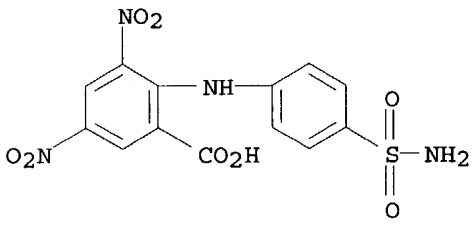
alc. 24 hrs. at 20°/4 atmospheric over 500 mg. PtO2, filtered, and the filtrate and alc. washings evaporated (N atmospheric) in vacuo to give the corresponding I [Ar = 2,4,6-HO2C(H2N)2C6H2, Ar' = p-C6H4SO2-NH2], decomposing on heating. VIII (500 mg.) hydrogenated and the filtered solution and 150 ml. PhNO2 washings combined, the alc. evaporated and the mixture refluxed 48 hrs., filtered and the filtrate concentrated in vacuo (0.1 mm.) gave 54% amorphous II (R = 8-NH2, R1 = 6-CO2H, R2 = 2-SO2NH2), m. >330. A similar synthesis employing o-H2NC6H4SO2NH2 led to II (R = 7-NH2, R1 = 9-CO2H, R2 = 1-SO2NH2) (IX) via I [Ar = 2,4,6-HO2C(O2N)2-C6H2, Ar' = 2-H2NSO2C6H4] (X). NaOAc (2.05 g.), 1.72 g. o-H2NC6H4SO2NH2, and 2.91 g. 2,3,5-Br(O2N)2C6H2CO2H refluxed 12 hrs. in AmOH with stirring and the yellow salt taken up in hot H2O, the solution boiled and the cold filtered solution acidified with HCl yielded 40% X, m. 220-2°. X (390 mg.) hydrogenated and the diamino compound oxidatively cyclized 44 hrs. in refluxing PhNO2 yielded 120 mg. IX, m. >330°. The mother liquors extracted into 2N NaOH and the Et2O-washed, decolorized (C), and filtered extract acidified with AcOH yielded 4% 3-aminophenazine-1-carboxylic acid. II (R = 3-NH2, R1 = 9-CO2Me, R2 = 1-SO2-NH2) (XI) was prepared from I [Ar = 2-HO2CC6H4, Ar' = 4,6,2-(O2N)2(H2NO2S)C6H2] (XII). ClSO3H (50 ml.) stirred 3 hrs. at 93° with 10 g. dry 2,3,5-Cl(O2N)2C6H2SO3Na and the cooled mixture poured onto ice yielded 55% 2,3,5-Cl(O2N)2C6H2SO2Cl, m. 104-6° (ligroine, b. 100-20°), converted by shaking with excess aqueous NH4OH to yield 84% 2,3,5-Cl(O2N)2C6H2SO2NH2, m. 198-209°. The crude sulfonamide (3.9 g.), 1.9 g. o-H2NC6H4-CO2H, and 2.9 g. NaOAc refluxed 4 hrs. in 100 ml. alc. with stirring and the red-orange Na salt taken up in warm H2O, acidified with concentrated HCl, and the free acid (2.45 g.) recrystd. from 95% alc. gave XII, m. 287-8°. XII (206 mg.) in 20 ml. alc. hydrogenated 24 hrs. at 20°/4 atmospheric over 206 mg. PtO2 and filtered, the residue extracted with hot alc. and the combined filtrate and washings evaporated (N atmospheric) in vacuo gave I [Ar = 2-HO2CC2H4, Ar' = 4,6,2-(H2N)2(H2NO2S)C6H2], m. 233.5-4.5° (H2O). XII (1.26 g.) in 30 ml. anhydrous MeOH containing dry HCl refluxed 8 hrs. yielded 75% I [Ar = 2-MeO2C6H4, Ar' = 4,6,2-(O2N)2(H2NO2S)C6H2] (XIII), m. 230.5-2.5°. XIII (100 mg.) in 10 ml. alc. hydrogenated 24 hrs. at 20°/4 atmospheric with 100 mg. PtO2 and the reduced product acetylated gave I [Ar = 2-MeO2CC6H4, Ar' = 4,6,2-(AcNH)2-(H2NO2S)C6H2], m. 232-3° (95% alc.). XIII (500 mg.) hydrogenated and cyclized 65 hrs. in refluxing PhNO2 gave 26% dark red XI, m. 276-7° (PhNO2). Chromatography of the mother liquors on Al2O3 and elution of the C6H6-washed column with 1:9 EtOH-Me2CO gave XI and 3-aminophenazine-1-sulfonamide (XIV). XII (360 mg.) hydrogenated and cyclized 44 hrs. in boiling PhNO2 gave 124 mg. impure II (R = 3-NH2, R1 = 9-CO2H, R2 = 1-SO2NH2) (XV), which was also obtained by oxidative cyclization of I [Ar = 2-HO2CC6H4, Ar' = 4,6,2-(H2N)2(SO2NH2)C6H2]. The mother liquors chromatographed on Al2O3 and the Et2O-washed column eluted with Me2CO gave XIV. XI (50.5 mg.) in 5 ml. 2N NaOH kept 30 min. at 100° and diluted to 15 ml., the filtered solution cooled and the pH adjusted to 5 by addition of AcOH yielded 95% XV, m. >330°. Oleum (15 ml., 20%) containing 2,3-Br(O2N)C6H3CO2H heated 3 hrs. at 155-60°, the cooled mixture added to a min. of ice, and the hot filtered solution salted out with NaCl gave 4,5,3-Br(O2N)(HO2C)C6H2-SO3Na (XVI), m. >300°. Oleum (90 ml., 20%) containing 25 g. o-BrC6H4CO2H heated 4 hrs. at 100°, cooled and treated below 40° with 25 ml. fuming HNO3 (d. 1.5), the mixture cautiously warmed to 98° and the temperature maintained 5 hrs., the cooled mixture poured onto ice and kept 16 hrs., filtered and salted out with NaCl yielded 91% XVI, converted by heating 3 hrs. at 96-8° in ClSO3H to yield 58% 4,5,3-Br(O2N)(HO2C)C6H2SO2Cl, m. 197-9°, stirred (8.8 g.) in 50 ml. aqueous NH4OH (d. 0.88) to give

6.6 g. 4,5,3-Br(O2N)(CO2H)C6H2SO2NH2 (XVII), m. 218-21°. XVII (6.5 g.), 3 g. p-H2NC6H4NHAc, and 4.1 g. NaOAc refluxed 4 hrs. in 50 ml. alc. and the residue on evaporation taken up in H2O, the filtered solution acidified with concentrated HCl and the precipitate recrystd. from dilute AcOH yielded 68% I [Ar = 4-AcNHC6H4, Ac' = 2,6,4-HO2C-(O2N)(H2NO2S)C6H2], m. 250-1°, hydrolyzed by refluxing 1 hr. in 2N HCl to give 68% I [Ar = 4-H2NC6H4, Ac' = 2,6,4-(HO2C)(O2N)(H2NO2S)C6H2] (XVIII)-HCl salt. The salt (500 mg.) in 20 ml. MeOH containing dry HCl refluxed 8 hrs. and the MeOH evaporated in vacuo, the residue taken up in cold HCl and the filtered solution treated with aqueous NaOAc gave 66% I [Ar = 4-H2-NC6H4, Ar' = 2,6,4-(MeO2C)(O2N)(H2NO2S)C6H2] (XIX), m. 193.5-6.5°. Attempted recrystn. of XIX from BuOH gave XVIII, m. 268-70°, converted into the above-mentioned HCl salt. XIX (300 mg.) hydrogenated and the product cyclized by boiling 60 hrs. in PhNO2, the cooled, filtered mixture chromatographed on Al2O3 and the washed (C6H6, Et2O, Me2CO, EtOH, H2O) column eluted with 1% aqueous C5H5N yielded 14% II (R = 8-NH2, R1 = 4-CO2H, R2 = 2-SO2NH2) (XX), m. >330°. Synthesis of XX via I [Ar = 2,4-(O2N)2C6H3, Ar' = 2-HO2CC3H4] (XXI) was attempted. ClSO3H (7.5 ml.) added slowly to 10.5 g. dry XXI with effervescence and rise of temperature to 70°, the mixture kept 1 hr. at 110° (oil bath), and the cooled mixture poured onto ice yielded 92% 5,7-dinitroacridone-2-sulfonyl chloride, m. 272-6° (decomposition) (PhMe), converted by addition of concentrated NH4OH to yield 95% 5,7-dinitroacridone-2-sulfonamide, m. >300°. The reactants of the above model compds. II with aqueous acid, together with other evidence led to the given structure for aeruginosin B (XXII), a red crystalline pigment from a strain of Pseudomonas aeruginosa.

IT **2379-43-3**, Anthranilic acid, 3,5-dinitro-N-(p-sulfamoylphenyl)-
2379-44-4, Benzoic acid, 3,5-diamino-2-(p-sulfamoylanilino)-
(preparation of)

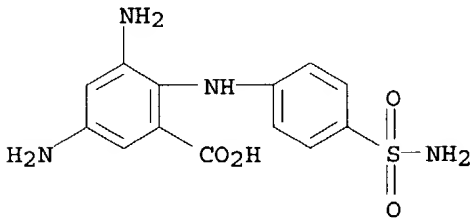
RN 2379-43-3 CAPLUS

CN Benzoic acid, 2-[[4-(aminosulfonyl)phenyl]amino]-3,5-dinitro- (9CI) (CA INDEX NAME)

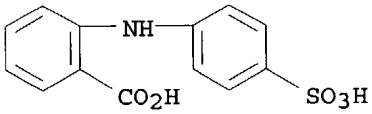


RN 2379-44-4 CAPLUS

CN Benzoic acid, 3,5-diamino-2-[[4-(aminosulfonyl)phenyl]amino]- (9CI) (CA INDEX NAME)



L5 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1955:23706 CAPLUS
DOCUMENT NUMBER: 49:23706
ORIGINAL REFERENCE NO.: 49:4558f
TITLE: New oxidation-reduction indicators. IV. Diphenylamine-4-sulfo-2'-carboxylic acid
AUTHOR(S): Cherkasov, V. M.
SOURCE: Zhurnal Obshchei Khimii (**1953**), 23, 201-3
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: English
AB See C.A. 48, 2655a.
IT **26119-52-8**, Anthranilic acid, N-p-sulfophenyl- (and derivs., as oxidation-reduction indicators)
RN 26119-52-8 CAPLUS
CN Benzoic acid, 2-[(4-sulfophenyl)amino]- (9CI) (CA INDEX NAME)



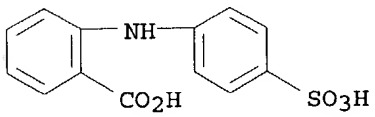
L5 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1954:14610 CAPLUS
DOCUMENT NUMBER: 48:14610
ORIGINAL REFERENCE NO.: 48:2655a-d
TITLE: New oxidation-reduction indicators. IV. Diphenylamine-4-sulfo-2'-carboxylic acid
AUTHOR(S): Cherkasov, V. M.
CORPORATE SOURCE: N. F. Gamale Epidemiol. and Microbiol. Inst., Dnepropetrovsk
SOURCE: Zhurnal Obshchei Khimii (**1953**), 23, 197-9
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C.A. 48, 645c. Heating 11.5 g. Na sulfanilate, 9.5 g. o-ClC6H4CO2Na, 0.1 g. powdered Cu, and 0.6 g. CuSO4 in sealed tubes with 20 ml. H2O 15 hrs. at 115-20° gave, after filtration, acidification with HCl to Congo red, and extraction of the precipitate with Et2O to remove o-ClC6H4CO2H, 28.8% diphenylamine-4-sulfo-2'-carboxylic acid in the form of the Ba salt, by precipitation of the filtrate with saturated BaCl2. The salt, (C13H10O5NS)2Ba, (5.2 g.) was heated on steam bath with 10 ml. H2O and 0.5 ml. concentrated H2SO4, filtered hot, adjusted to 20 ml., and treated with 7 ml. HCl (d. 1.19); the product is least soluble in 20% HCl, and repptn. as above gave the pure acid (I), greenish plates, decompose without melting. I with BaCO3 yields a water-soluble Ba salt, C13H9O5NSBa. I (2.93 g.) in 5 ml. H2O with 0.53 g. Na2CO3 gave C13H10O5NSNa.H2O, poorly soluble in H2O, insol. in EtOH. I (2.93 g.) treated in 10 ml. H2O with 1.06 g. Na2CO3 and 5 ml. H2O, then evaporated, gave very soluble C13H9O5NSNa2. I (1.46 g.) in 15 ml. 10% NaOH treated with 5.04 g. Me2SO4 and acidified after 15 min. gave the mono-Me ester, which ppts. on acidification; it is soluble in EtOH. The Ba salt is sparingly soluble in H2O. The ester decompose

before melting. I (0.001M solution) used as an indicator in titrations with K2Cr2O7 and Ce sulfate showed a color change from colorless to yellow-green, finally to pure blue-violet. In 10N H2SO4 the least amount of the oxidizing agent (0.001N) is 2 ml.; the optimum amount is 4 ml. At lower acidity than 10N H2SO4 the color change is delayed. If Fe++ is present the color change occurs even in 7.5N H2SO4. The mono-Me ester has similar indicator properties.

IT **26119-52-8**, Anthranilic acid, N-p-sulfophenyl-
(and derivs., as oxidation-reduction indicators)

RN 26119-52-8 CAPLUS

CN Benzoic acid, 2-[(4-sulfophenyl)amino]- (9CI) (CA INDEX NAME)



=> LOG Y

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	52.45	208.08

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE ENTRY	TOTAL SESSION
CA SUBSCRIBER PRICE	-6.93	-6.93

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